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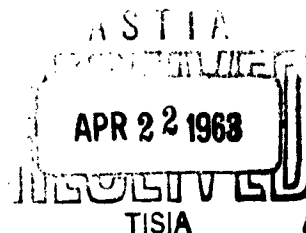
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**SOLID SOLUTIONS IN
COPPER-IRON ALLOYS**

WILLIAM KLEMENT JR.

MARCH 1963

**A REPORT ON RESEARCH CONDUCTED UNDER
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SOLID SOLUTIONS IN COPPER-IRON ALLOYS

by
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Technical Report No. 13 submitted to:

U. S. Atomic Energy Commission Contract No. AT(04-3)-221

Office of Naval Research, Contract No. Nonr-220(30)

Approved by Pol Duwez
Professor of Engineering

March 1963

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ABSTRACT

By rapidly quenching liquid alloys, single-phase copper-rich solid solutions are obtained from 0-20 at. pct. Fe. The plot of lattice spacings vs. composition exhibits a distinct maximum near 7.2 ± 0.5 at. pct. Fe and this extremum is tentatively associated with a magnetic transition. Body-centered cubic iron-rich phases containing about 5-10 at. pct. copper coexist with face-centered cubic copper-rich phases of rather widely varying compositions in the range, 20 - about 80 at. pct. Fe. At least 10 at. pct. Cu is retained in solid solution in the iron-rich phase, for which the lattice spacing increases by $1.1 \pm 0.1 \times 10^{-3}$ Å per at. pct. Cu.

1. INTRODUCTION

By rapidly cooling alloys from the melt, metastable solid solutions can often be obtained for components of similar crystal structure which normally form eutectic or peritectic systems. The inability to achieve a continuous series of single-phase solid solutions in Cu-Co alloys by quenching in this way⁽¹⁾ may be related to the rapid segregation occurring within the miscibility gap in the undercooled melts. In the present investigation, Cu-Fe alloys were quenched from the melt and examined by means of x-ray diffraction in order to determine the extent of solid solubilities and to further delineate the role of the miscibility gap (Fig. 1) in the solidification process.

Lattice spacings of the copper-rich face-centered cubic (fcc) solid solutions were determined with the Debye-Scherrer technique and compared with previous work⁽⁵⁾. Similarly, lattice spacings were obtained for the iron-rich body-centered cubic (bcc) solid solutions resulting from the diffusionless $\gamma \rightarrow \alpha$ transformation.

2. EXPERIMENTAL PROCEDURES

Alloys were prepared from elements of greater than 99.9% purity. Some of the copper-rich alloys were cast into wires but most of the alloys were prepared from reduced powders that were mixed 24-120 hr, pressed into compacts that were sintered at 1000°C for 24-48 hr under hydrogen and then furnace cooled. The methods of alloy preparation were very similar to those described for the Cu-Co investigation⁽¹⁾, as were the quenching techniques. Because of the greater reactivity of iron, the iron-rich alloys were liberally covered with argon while at elevated temperatures.

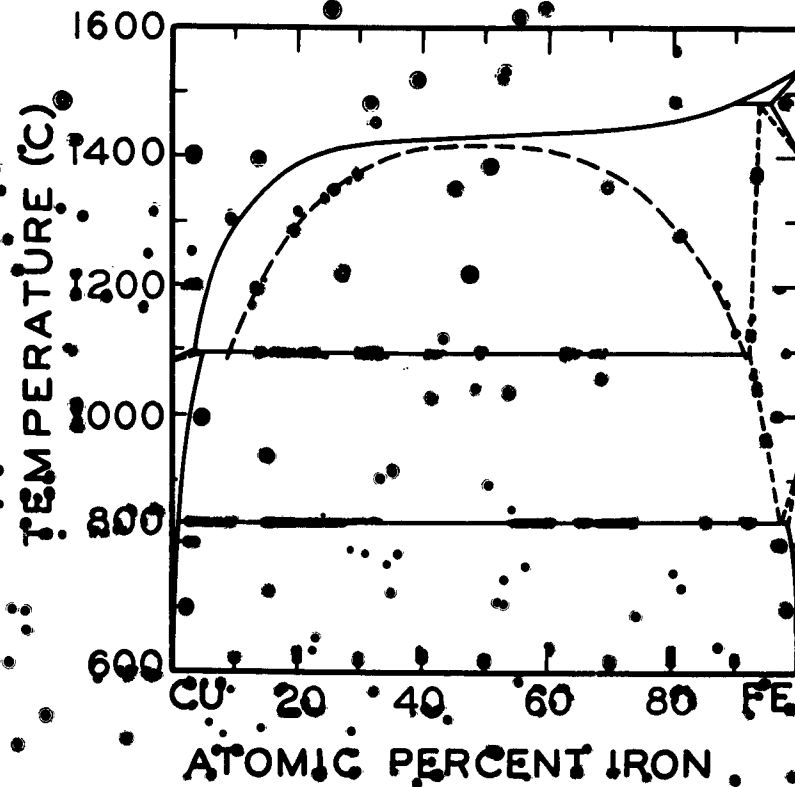


Figure 1. Equilibrium phase diagram for the Cu-Fe system, after Hansen and Anderko⁽²⁾, with the ferrite boundary determined by Wriedt and Darken⁽³⁾. The metastable miscibility gap has been calculated from the data and model of Nakagawa⁽⁴⁾.

The flakes obtained for the quenched iron-rich alloys were invariably less than 1 mm^2 in area, with slightly larger flakes obtained for the copper-rich alloys. The x-ray procedures and Debye-Scherrer film work were much the same as previously described⁽¹⁾⁽⁶⁾⁽⁷⁾. Nickel-filtered CuK radiation ($\lambda(\text{CuK}\alpha) = 1.54178 \text{ \AA}$, $\lambda(\text{CuK}\alpha_1) = 1.54050 \text{ \AA}$) was used for the alloys containing less than 65. at. pct. Fe; iron-filtered CoK radiation ($\lambda(\text{CoK}\alpha) = 1.79020 \text{ \AA}$) was used for the alloys of greater iron content. All work was done at room temperature, $25 \pm 2^\circ \text{C}$.

EXPERIMENTAL RESULTS

The relative visual intensities of the phases detected on the films of the quenched alloys are presented in Table 1, with the intensity of the predominant phase arbitrarily taken as "strong". The lattice spacings of the fcc structures detected in the alloys containing up to 80. at. pct. Fe are plotted in Figure 2, together with the data of Anderson and Kingsbury⁽⁸⁾ and the discordant datum of Hutcheson and Reekie⁽⁵⁾. High angle diffraction lines were resolved for the fcc structures in alloys containing up to 20.0 at. pct. Fe; lines for all other structures in the quenched alloys were not resolved.

For the alloys containing more than 80. at. pct. Fe, fcc phases with lattice spacings between 9.61, and 9.63 \AA were occasionally detected. However, there was no observable difference in the lattice spacings of the predominant phases with these fcc phases present or absent. The lattice parameters for the bcc structures are:

composition (at.pct. Fe)	lattice parameter (\AA)
95.	2.8720 ± 10
92.5	2.8745 ± 10
90.	2.8770 ± 10
85.	2.8810 ± 15
80. 75.	2.8770 ± 2
65.	2.876 ± 2

Table 1. Relative visual intensities of phases detected in quenched Cu-Fe alloys.

Composition (at. pct. Fe)	structures	
1.5, 2.5, 4.5, 6.0, 6.5, 7.2, 8.0, 8.5, 10.0, 10.5, 12.0, 15.0, 17.0 18.5, 20.0*	fcc	bcc
25., 35., 45., 55.	stf	vw
65.	stf	med
75., 80.	v	stf
85., 90., 92., 95.	stf	stf

*These alloys, ± 0.5 at. pct.; other alloys, ± 1 at. pct.

**Fcc structures, herein assigned to contamination, occasionally detected for these quenched alloys (cf. text).

stf = strong, med. = medium, vw = very weak.

For pure, quenched iron, $a = 2.8665 \pm 8 \text{ \AA}$, with the high angle lines resolved. Lattice spacings for the minority bcc phases in the alloys of higher copper content ranged from 2.87_0 to 2.87_6 \AA . All lattice spacings are from three or more independent determinations and are considered quite reproducible except for the aforementioned fcc phases in the iron-rich alloys.

4. DISCUSSION

4.1 Solidification and the Metastable Miscibility Gap

Several of the mechanisms involved in the metastable extensions of solid solubility or in the occasional retention of nonequilibrium phases by the rapid cooling of liquid alloys have been discussed previously⁽¹⁾⁽⁶⁾⁽⁹⁾. Of greatest pertinence here are the experiments⁽¹⁾ with Cu-Fe alloys, which clearly demonstrated the need for considering the segregation processes occurring within the miscibility gap in the undercooled melt.

A portion of the boundary of the miscibility gap for undercooled Cu-Fe liquid alloys has been directly determined by Nakagawa⁽⁴⁾ and, as shown in Fig. 1, the remainder has been estimated to below the peritectic temperature according to the equation⁽⁴⁾.

$$T(^{\circ}\text{K}) = \frac{3720 (1-2c)}{\log \left(\frac{1-c}{c} \right)} \left\{ 9.911 + 0.178 (1-2c)^2 \right\}$$

where c is the atomic fraction of copper or iron.

If the diffusion processes in the liquid alloys are as rapid as might be expected from the data⁽¹⁰⁾ for some elements, microscopic segregation within the miscibility gap would not be suppressed even with the very high cooling rates employed in the present experiments. It could then be suggested

that single-phase copper-rich solid solutions would not be obtained for alloys with an iron concentration greater than that fixed by the intersection of the gap boundary and the peritectic temperature - about 9 at. pct. Fe. Single-phase copper-rich solid solutions were obtained to about 20 at. pct. Fe, however, and this reasoning is perforce invalid if the miscibility gap boundary is reliable. It does not appear that much information about the postulated segregation within the miscibility gap can be deduced from the iron-rich alloys because of the diffusionless solid state transformation. Since the miscibility gap is approximately symmetrical, reasoning as above suggests that single-phase solid solutions would not be obtained from the melt for alloys containing less than about 91 at. pct. Fe. A much better correlation between the limits of metastable single-phase solid solubility and the gap-peritectic temperature intersections was found for the Cu-Co alloys⁽¹⁾ than for the Cu-Fe system.

For the quenched alloys of intermediate iron concentration, there is much similarity with the data from the Cu-Co investigation⁽¹⁾, i.e., there is one phase of relatively constant lattice spacing (and hence composition) and another phase of varying composition. For the Cu-Fe alloys, the iron-rich solid solutions contain about 5-10 at. pct. Fe within the two-phase region; on the other hand, for the Cu-Co alloys, the copper-rich phase was found to vary only slightly in composition. In order to account for the Cu-Co results, it was suggested⁽¹⁾ that copper-rich clusters containing about 10 at. pct. Co separated out preferentially during the undercooling into the miscibility gap. By analogy, iron-rich clusters containing less than about 10 at. pct. Cu may preferentially segregate during the undercooling, then solidify and transform to bcc upon further cooling.

4.2 Lattice spacings of the fcc solid solutions

The variation of the present lattice spacings with composition (Fig. 2) for the copper-rich alloys is in good agreement with the results of Andersen and Kingsbury⁽⁸⁾, although there is some disagreement in absolute value. This discrepancy may be due to the different temperatures at which the x-ray measurements were carried out and perhaps to the different extrapolation procedures*. In his comprehensive compilation⁽⁵⁾, Pearson expresses some surprise that solution of iron increases the lattice spacing of copper but nevertheless recommends the Andersen-Kingsbury results to the datum of Hutchison and Reekie⁽⁵⁾ because the former's alloys are more nearly in equilibrium. This would appear to be an irrelevant criterion here since the present quenched alloys are hardly in equilibrium. Another lattice spacing investigation of the copper-rich iron alloys was carried out by Bradley and Goldschmidt⁽¹¹⁾. Despite some data to the contrary, these workers also concluded that the lattice parameter of copper is initially decreased by the solution of iron.

Some workers⁽¹²⁾ have linearly extrapolated lattice spacings of dilute alloys to 100% solute in an attempt to obtain the "apparent size" of the solute. Such an extrapolation of the present results or of the Andersen-Kingsbury data would yield an apparent lattice parameter of fcc iron at room temperature of about 3.67-3.68 Å -- somewhat above estimates from other sources, as discussed below. The occurrence of the distinct maximum near 7.2 at. pct. Fe in the plot of lattice spacing vs. composition (Fig. 2) suggests, at the very least, that considerable caution should be exercised in any reasoning based on these sorts of extrapolations.

*Andersen and Kingsbury state that their lattice spacings were computed relative to the wave lengths given by Siegbahn in 1933; this reference has not been found and it is assumed that the well-known 1931 wavelengths were used, the lattice spacings then being in kX units which are converted into Å by the factor 1.00202.

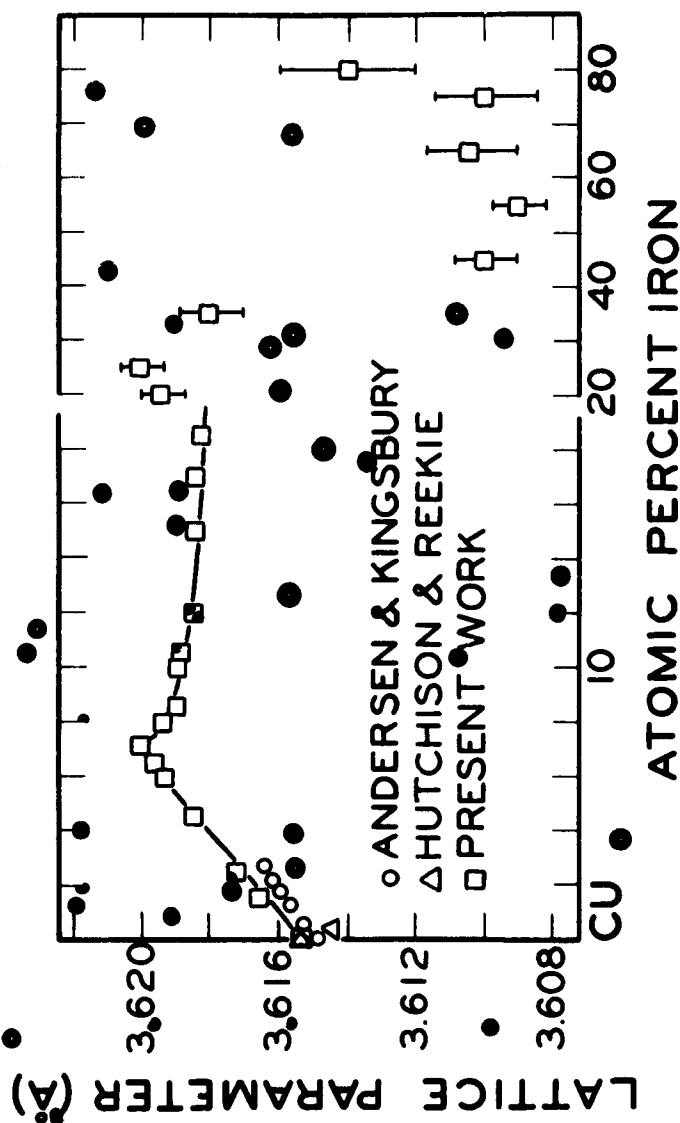


Figure 2. Lattice spacings of the fcc solid solutions in Cu-Fe alloys. The data for the 20.-80. at. pct. Fe alloys are from two-phase specimens. Uncertainties in the present lattice parameters are indicated by the dimensions of the symbols or by the error bars.

The maximum in the lattice spacing vs. composition curve near 7.2 ± 0.5 at. pct. Fe (Fig. 2) is believed to be associated with a change in the magnetic character of the solid solutions. Bitter, et al.⁽¹³⁾ and Scheil, et al.⁽¹⁴⁾ have measured the susceptibilities of some single-phase copper-rich iron alloys and determined the "Curie" temperatures. Direct measurements below the second-order transformation do not seem to have been made and it is by no means clear that the solid solutions are ferromagnetic rather than antiferromagnetic - in cognizance of the recent proposal⁽¹⁵⁾ for γ -Fe. The plot of Curie or Néel temperature vs. composition given by Scheil, et al.⁽¹⁴⁾ may be extrapolated with an intersection near room temperature and 7.2 at. pct. Fe not unlikely. Other extrema in lattice spacings⁽⁵⁾ found for para- to ferro- or antiferromagnetic alloys do not seem to be nearly as pronounced as for the present Cu-Fe data.

At iron concentrations greater than that of the extremum, the fcc lattice spacings of the single phase solid solutions gradually decrease. The lattice parameters of the fcc solid solutions found in the two-phase region, 20.-80. at. pct. Fe, are believed to be greater than the lattice parameters of the single-phase fcc solid solution for the given composition, if this could be obtained.

4.3 Lattice parameters of γ -Fe

Perhaps the closest approach to a direct estimate of the lattice spacing of fcc iron below its range of stability is that due to Newkirk⁽¹⁶⁾, who suggested $a = 3.588 \text{ \AA}$ at room temperature. This value was derived from the experimental datum by making a small but arbitrary correction for the copper in solid solution and also is apparently subject to some poorly understood "coherency" effects. Koritke⁽¹⁷⁾ has shown, however, that the epitaxial effects

for iron vapor-deposited onto a single-crystal of copper extended only to a thickness of $\sim 80 \text{ \AA}$, with no discernable epitaxy for nickel deposited in a similar way. Newkirk⁽¹⁶⁾ estimated the size of the γ -Fe particles as $\sim 1000 \text{ \AA}$ and it may be that dilatation due to the copper matrix does not extend over the bulk of the fcc iron-rich precipitates. If the "coherency" effects are tentatively rationalized away in this fashion, it still remains to account for the copper retained in solid solution. Newkirk⁽¹⁶⁾ assumed that about 3-4 at. pct. Cu was dissolved in the γ -Fe particles and adjusted the observed lattice spacing of 3.590 \AA to 3.588 \AA on the basis of an assumed linear variation in lattice parameter between copper and iron. The present results suggest that there is a positive deviation from linearity, perhaps even to the iron-rich end, and that a further, slight downward revision of the proposed γ -Fe lattice spacing may be necessary. It should be realized, however, that the amount of copper in solid solution in these precipitates is not actually known. Although the effect on the lattice spacing cannot be too great, such physical parameters as the Neél temperature might be greatly affected. In fact, it may be suggested that the Neél temperature of 8°K proposed for γ -Fe, on the basis of experiments by Abrahams, et al.⁽¹⁵⁾ with iron-rich precipitates in a copper matrix, be regarded only as tentative until the effects of the alloying are isolated. It would be useful to compare the 8°K value with extrapolations from austenitic phases, but no systematic values are available.

In the same vein, any estimates of the lattice spacings of γ -Fe must be consistent with extrapolations from austenitic phases. According to the data collected by Pearson⁽⁵⁾ for several substitutional and interstitial solid solutions, the value for γ -Fe by extrapolation is $\sim 3.578 \text{ \AA}$ at room temperature.

Several sets of Fe-C lattice parameters⁽¹⁸⁾ lead to somewhat lower value, however, and the situation is ambiguous. Jamieson⁽¹⁹⁾ attempted to resolve the quandry by suggesting that several values for fcc iron were possible. Kaufman, et al.⁽²⁰⁾ have vigorously developed this proposal and also suggested correlations between volume and magnetic moment/atom. The arguments of Kaufman, et al.⁽²⁰⁾ employ extrapolations to $a \sim 3.64 \text{ \AA}$, from Pt-, Pd- and Ni-Fe alloys, which do not appear especially convincing to the present writer.

Lattice parameter extrapolations are improved if the deviations from linearity can be taken into account. Klement⁽¹⁾ has proposed a dimensionless "distortion" parameter which empirically seems to offer some correlations between the deviations from linearity and the positions of the components in the periodic classification. Klement and Luo⁽²¹⁾ have shown that rather good correlations obtain for Pd- and Pt- base binary alloys, except that anomalies due presumably to magnetic effects enter in a way which, so far, is not understood. Calculations of the distortion parameter vs. composition carried out for fcc binary alloys⁽⁵⁾ with iron seem to indicate a good deal of complexity -- much of which may be tentatively ascribed to magnetic effects. The only correlations apparent involve the distortions for the solute-rich alloys of Fe with the transition elements of the first long period, for which the distortions proceed from positive to negative in the sequence, Cu \rightarrow Ni \rightarrow Co \rightarrow Fe \rightarrow Mn.

4.4 Iron-rich alloys

The variation in lattice spacing with composition for the bcc structures is approximately linear, with a slope of $1.1 \pm 0.1 \times 10^{-3} \text{ \AA/at. pct. Cu}$, up to at least 10 at. pct. copper. This slope is in good agreement with the

value of 9.4×10^{-3} Å/wt. pct. Cu⁽³⁾ found for some dilute alloys. If the reasonable suggestion is allowed that the bcc lattice spacing vs. composition plot deviates somewhat from linearity, perhaps up to 15 at. pct. copper might have been dissolved. For the alloys of higher copper content, it is clear, however, that there has not been a diffusionless transformation, without change in composition, from a single-phase fcc solid solution.

Recent work⁽²²⁾ suggests that the fcc \rightarrow bcc transformations in these iron-rich alloys should be martensitic rather than massive at the present, very high cooling rates. This, of course, could not be verified because of the inconveniently small and fragmentary specimens. The transformation mechanism may, in fact, have been greatly altered due to the possible contamination with carbon and/or nitrogen.

As mentioned elsewhere, fcc phases occasionally were detected in the quenched iron-rich alloys. The range of lattice spacings, although not reproducible, suggests⁽⁵⁾ that nitrogen and/or carbon-stabilized austenite had been formed. The interstitial solid solubility of carbon and nitrogen in the bcc structures is low⁽⁵⁾ and probably cannot be metastably extended very much because of the ease of diffusion and hence does not affect⁽⁵⁾ the bcc lattice spacings significantly. The high degree of reproducibility of the lattice spacings for the alloys containing less than 80 at. pct. Fe strongly suggests that contamination was unimportant for these alloys.

The appreciable re-engineering of the apparatus necessary to definitely exclude the possibility of carbon and nitrogen pickup was not undertaken for at least one reason. After the present experiments were in progress, a paper⁽²³⁾ by Kneller appeared which reported the success in obtaining a continuous series of solid solutions for Cu-Co alloys by vapor deposition.

This technique should be easily extensible to Cu-Fe alloys, with little contamination problems, and with the very promising prospect that foils of a size suitable for measurement of many physical properties might be obtained. The difficulties in the liquid \rightarrow solid transformation would also be avoided and it is likely that the solid solubilities could be extended beyond those reported in this paper.

ACKNOWLEDGEMENTS

Thanks are due to Dr. P. A. Flinn and H. L. Luo for their comments on the manuscript. J. H. McCoy and P. Knust-Graichen assisted considerably with the experiments.

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